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SIZE EFFECT IN YIELD STRENGTH
IN ALUMINUM SINGLE CRYSTALS

by

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Although much research has been devoted to the effect of surface conditions on the mechanical properties of metal single crystals, the question of size effect with and without surface films has been rather sparsely considered experimentally. The Griffith crack theory(1) was quite successful in explaining size effect in glass and, before the advent of dislocation theory, was considered adequate in explaining size effect in metals. The success of the dislocation theory in explaining, among other phenomena, the low value of yield stress, as compared with the theoretical strength, has displaced the Griffith crack theory as applied to metals.

Very good reviews of the effect of surface conditions on properties of metal crystals exist: among these the recent "Symposium on Properties of Metallic Surfaces" of the Institute of Metals(2) and the compilation by Brown(3) should be noted. Of the research on size effect in single crystals, perhaps one of the earliest was that of Ono(4) who pulled single crystals of aluminum of various sizes. Later work by Maddin and Hibbard(5) on alpha brass single crystals was incomplete since the crystals varied from 13 mm. to only 3 mm. on an edge. Despite the insufficient variation in cross-section, a small size effect was noted. More recently, Makin(6) extended single crystals of cadmium, both clean and oxidized, from 1.1 to 0.025 mm. in diameter. Here a definite size effect was noted both for the clean and for the oxidized crystals.

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(1) References will be found at the end of the paper.

Since seeding techniques for growing crystals have provided relatively easy methods for growing different size crystals with the same orientation, it was considered desirable to investigate the effect of size and its relation to the shear along glide planes.

EXPERIMENTAL PROCEDURE

Large Specimens

Aluminum, 99.997 per cent pure, supplied through the courtesy of the Aluminum Company of America was converted into single crystals 12 inches long by 1/16 inch square up to 10/16 inch square in 1/16 inch intervals having the same orientation. The mold consisted of a sectioned high-purity graphite rod in which there were milled ten square holes with a common sink at the bottom of the assembly. Molten aluminum at 870°C was poured into the mold held at the same temperature and cooled from the bottom by means of a cooling block.

The rods were etched in Aqua Regia, cut into six inch lengths and x-rayed to determine their orientation. Only those with identical orientations were chosen for the tensile tests. The center three inch section of each specimen was mechanically polished through 400 emery, etched and homogenised for 24 hours at 600°C. Following the homogenization treatment, they were electrolytically polished (1:2 HNO_3 - CH_3OH solution) and finally chemically polished for 15 minutes in ALCOA R-5 bright dip. All dimensioning of cross-sections was made just prior to the last five minute bright dip.

An edge of each of the specimens to be tested was fitted with a Baldwin SR-4 type A-8 strain gage held with Duco cement; the specimens were pulled to a constant stress of 580 psi (411 g/mm²) in an Olsen Plastiversal machine at a strain rate of 0.005 per second.

Interferometric measurements of shear were made on two adjacent sides of the specimens from representative interferograms. Slip line counts were

made at x 200 and averaged from at least three individual counts on adjacent faces on each specimen.

Small Specimens

Because of the ease with which small diameter single crystals of aluminum may be damaged, it was necessary to use a special apparatus to determine the "yield". A sketch of the apparatus is shown in Fig. 1. The specimen was allowed to slide in a slotted bakelite block. One end of the specimen was polished down to a small diameter and soldered in place inside a drilled-out soldering pencil. A spring hook clamped to the other end, was connected to a string from which a connected pan could be loaded with weights. The assembly was mounted on the stage of a microscope. Motion of the specimen was measured with a calibrated eye piece.

The small specimens were prepared by electrolytically polishing a 1/16 inch square single crystal down to the desired diameter (from 90 to 400 microns), after which they were bright dipped from 5 to 15 minutes. The polishing caused the specimens to assume a necked appearance as shown in Fig. 1. The minimum diameter of the neck was measured in two positions at right angles to each other and the average used to compute the cross sectional area.

The use of necked specimens has some disadvantages but there was no simple way to secure a specimen of uniform cross sectional area in the range 10^{-1} to 10^{-2} mm^2 while still making use of the 1/16 inch square crystal whose orientation was the same as that of the larger crystals. The friction of the apparatus could be determined after the specimen had fractured.

Results

The series of large crystals designated as MP were extended at a constant strain rate of 0.005 per second to a constant stress of 580 psi (411 gm/mm^2). The series of stress-strain curves is shown in Fig. 2. Since no sharp yield was noted for these crystals an arbitrary "yield" was defined as the projection of the linear section in the plastic region back to an intersection with the ordinate. In the case of specimen MP 3/16 which was not extended well into the plastic region, the stress-strain curve was extrapolated somewhat. The uncertainty of the yield point for MP 3/16 is consequently greater than for the other large specimens. If one makes the assumption that the slopes of the linear portions of the stress-strain curves in the plastic region are all about the same, then it is clear that the "yield stress" of MP 3/16 is greater than that of the other large specimens. The difference in the amount of elongation as compared with the size of the specimen is particularly interesting in that it agrees qualitatively with the earlier observations on alpha brass(5).

For the small necked specimens the stress is greatest at the neck and the strain consequently varies along the specimen axis. It is not possible to relate the extension observed at a given load to the strain at a certain position in the specimen without a detailed knowledge of the specimen shape and some involved calculations. Therefore only load-extension data are plotted for these specimens. The linear portion of the load-extension curve is projected back to the ordinate and this load divided by the minimum area at the neck before loading is taken to be the "yield stress". Figs. 4, 5 and 6 are the load-extension curves for the small specimens used. The uncertainty of "yield stress" for these small specimens is estimated to be ± 10% due to lack of precise knowledge of the cross sectional shape.

Table I gives the stress at "yield" for the various specimens.

TABLE I

Spec. No.	Stress at "Yield" gm/mm^2	Resolved Stress gm/mm^2	Area mm^2
MP 1/2	205	98.5	148
MP 5/16	282	136	55.6
MP 1/4	322	155	33
MP 3/16	400	192	20.8
MG-M	1600	737	0.16
MG-S	3300	1480	0.036
MG-W	3700	1660	0.026

Fig. 7 is a plot of the log of the yield stress versus the log of the cross sectional area. Within the accuracy of the measurements, the "yield stress" is inversely proportional to the 1/3 power of the area. Initial orientations of the specimens extended are shown in Fig. 8.

The number of slip bands was counted on adjacent faces opposite the strain gage for the gage length of 1/8" and also 1/8" on either side of the gage length. The average from a minimum of three counts was recorded. The average shear height, s, was determined by interferometric techniques and from this h, the contribution of the band to the elongation, was calculated. The total elongation measured interferometrically is the product of the average number of slip bands by the average contribution of a slip band to the elongation. Table II compares the interferometrically measured elongation Δl_i with the strain gage measured elongation Δl_s for various sized specimens all pulled to <u>constant stress</u>.

TABLE II

Spec. No.	Average No. of Slip Bands per 1/8"	Average Shear Height s(Å)	h Å	Δl_1 Å	Δl_s Å
3/16	57	530	524	29800	4100
1/4	48	540	530	25500	15000
5/16	61	520	510	30900	35000
1/2	57	455	450	25600	38800

Discussion

The data in Table I seem to indicate that a size effect in yield strength exists. However, the data should be considered tentative for there are several effects whose importance has not as yet been ascertained that might cause much of the effect to disappear. For the large specimens, strain was measured by means of a resistance type strain gage which was cemented to the crystal surface. (The gage has a paper base to which the resistance wire is bonded). The force necessary to extend the cemented strain gage will be a relatively larger fraction of the applied force for the smaller crystals. The cement might also act as a tough skin which would interfere with the normal gliding action of the lamellae during deformation. This would raise the yield strength as the fixed area of the gage would cover more of the surface area of the small crystals than of the large ones.

Another question about the data obtained on the series of large crystals is the fact that only the two largest crystals showed the same modulus of elasticity (19×10^6 psi) and no specimen showed the correct modulus. The two other crystals showed much lower moduli: 7×10^6 psi for the 1/4" crystal and 5×10^6 psi for the 3/16" crystal. Since the modulus of elasticity of aluminum varies only between the limits of 6.4×10^6 and 7.7×10^6 g/mm^2 (9.1×10^6 and 1.09×10^7 psi) regardless of orientation according to Barrett[7], a serious systematic error in the experiment is indicated. There is no necessary relationship between the failure to get the correct

modulus of elasticity and the size effect of yield point, but it would certainly be in order to determine the cause of this variation of the moduli. Possibly an error in the strain scale of Fig. 2 exists.

As mentioned above, the use of necked specimens was dictated by necessity. The most serious defect in using necked specimens probably is the variation of shearing stress along the slip plane. If the slip plane makes a very acute angle with the stress axis, the slip plane may have some regions of very low shearing stress and other regions with high shearing stress. It is not clear how the data from necked specimens relates to the data from specimens with uniform cross section. It is shown in Appendix I that a parabolic relationship between stress and strain is only modified by a constant factor when dealing with necked specimens. However, the derivation applies only to the case where differential volume elements can be considered to be unconnected by such large scale weaknesses as slip planes. In other words, the derivation would apply to a necked polycrystalline specimen but not to a necked single crystal specimen.

If the effects of the cemented strain gage prove to be negligible, the size effect in yield strength is of somewhat surprising magnitude in view of the results of other investigators(6) (8). Makin studied the strength of cadmium crystals from 1.1 mm diameter down to 0.025 mm diameter, both with and without an oxide film. His results are shown in Fig. 7 and apparently the critical shear stress is independent of size above 0.7 mm diameter. For the aluminum crystals reported here, there is a measurable increase in yield stress between the 5/16" square (8.0 mm) and the 1/2" square (12.8 mm) crystals. The critical shear stress for Makin's smallest specimen (0.025 mm diameter) is only a factor of 3 or so times that of the largest specimens, while this data shows a factor of 18 from the smallest (0.16 mm diameter) to the largest (1/2" square). However, cadmium has a hexagonal close-packed

structure, while aluminum has a face centered cubic structure, and one should not expect identical behavior from both metals.

The unavoidable presence of an oxide coating on every aluminum specimen immediately raises the question of how much of the apparent size effect is attributable to the oxide and how much to the aluminum. Aluminum oxide has an enormous compressive strength (700 kg/mm^2) and, if its tensile strength is at all of the same order of magnitude, the oxide might be expected to contribute considerable tensile strength to a small specimen. That this is unlikely can be shown by a rough calculation. Aluminum oxide has a modulus of elasticity of about 14×10^6 psi (10^7 gm/mm^2); and, if it has a high yield stress in tension (say 10^5 gm/mm^2), the yield point would not be reached until strains of the order of 10^{-2} or 1%. At strains of 10^{-4}, however, the specimen has already undergone extensive slip which would break the oxide film in many places.

The oxide film may have an important influence by acting as a barrier to the movement of dislocations. In order to cause a size effect in yield strength, some means could be postulated by which more dislocations are piled up at the oxide coating of a large crystal than for a small crystal for a given applied stress. Doubtlessly many mechanisms can be devised to accomplish this end. One possibility is to assume that there is a constant density of unlocked dislocations in a crystal. Under an applied stress, these dislocations would move to the oxide barrier. In a large crystal there would be a greater probability of getting a large number of dislocations piled up along one slip plane; and, since the stress in front of n dislocations is n times the applied stress, the large crystals might be expected to yield at a lower applied stress. Another possibility of piling up more dislocations at the surface of a crystal is to note after Mott[9] the length of

slip plane available for storing dislocations is larger in a large specimen than in a small one, on the average. The formula of Eshelby, Frank and Nabarro[10] gives the stress σ needed to pile up n dislocations against a barrier. The n dislocations will be confined to a distance L along the slip plane.

$$L = \frac{n \mu b}{\pi \sigma k}$$

and μ = shear modulus, b is the Burger's vector, k = numerical constant near unity.

For a given applied stress, the ratio of n to L is constant regardless of specimen size. Since the distance L is more likely to be large in a large crystal, a given applied stress will pile up more dislocations against the oxide barrier in a large specimen on the average. Mott has shown[9] that the length L to hold 1000 dislocations in an aluminum single crystal for a typical stress is 0.05 cm which implies that a size effect in yield strength is possible in the .1 mm to 1 mm range. This second mechanism would use a Frank-Read source to generate dislocations and would avoid the difficulties in the unlocked dislocation argument in getting sufficient numbers of dislocations on one slip plane to function as a large stress concentration factor. In view of the uncertainty of the data, further speculation on the size effect in yield strength serves little purpose.

The data of Table II are difficult to understand. The two largest crystals show an interferometrically measured elongation Δl_i which is less than Δl_g, the elongation deduced from the strain gage. This is reasonable since there is strong evidence to support the hypothesis of fine slip[11][12][13], i.e., slip below the limit of resolution of the multiple beam interferometer and the electron microscope. (The interferometer used can detect differences in height of 100 Å or more). The different methods of measuring elongation agree within 15% for the 5/16" crystal and 35% for the 1/2"

crystal. This is in qualitative accord with the results of Wilsdorf and Kuhlman-Wilsdorf who found 10% fine slip at 4% strain[11].

For the two smaller crystals, 1/4" and 3/16", the interferometrically measured elongation is greater than the strain gage measured elongation and the difference is not a small (10% approx.) effect. For the 1/4" crystal Δl_i is 29800 Å while Δl_g is 4100 Å. For the 3/16" crystal Δl_i is 25500 Å while Δl_g is 15000 Å. At present no explanation is offered for this result. The experiment should be repeated with a type of strain gage which causes much less interference.

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APPENDIX I

The usual practice in determining the stress-strain relationship with the use of a specimen of uniform cross-sectional area appeared too difficult to apply to single crystals in the 100 to 400 micron diameter range. The easiest method of performing the experiments appeared to be the one outlined in the text which uses necked specimens. The question immediately arises as to the functional relationship between stress and strain (or load and extension) for specimens of varying cross-section as compared to that for a specimen of uniform cross-section. It can be shown that for a linear stress-strain relationship, the load-extension curve of a specimen of varying cross-section will also be linear. If the stress-strain curve has a horizontal portion then the load-extension curve of a specimen of non-uniform cross-section will not show a horizontal segment unless the specimen has an appreciable length of constant cross-section. The stress-strain curves of the small aluminum crystals show a parabolic relationship between stress and strains, i.e. stress is porportional to the square root of the strain. As shown below, the assumption of a parabolic relationship between stress and strain produces a parabolic relationship between load and extension for a necked specimen.

Consider the appearance of a typical specimen as shown schematically in Fig. 11. Because the cross-sectional areas A_1 and A_2 away from the neck are 50 to 100 times that of A_o, the area of the neck, it may be assumed that the entire extension of the specimen occurs at the necked portion in the length marked L_o. A fair approximation to the manner in which the radius of cross-section at the neck varies along the specimen axis is a hyperbola. Fig. 12 shows the range of shapes encountered in these experiments. For each infinitesimal length of specimen dx the stress $\frac{P}{A(x)}$ will cause a second order infinitesimal increase in length d^2x. For a parabolic stress-strain

relationship we have

$$\frac{F}{A} = k\left(\frac{d^2x}{dx}\right)^{\frac{1}{2}} \qquad A = \pi y^2 = \pi(a^2+b^2x^2)^{\frac{1}{2}}$$

$$\frac{d^2x}{dx} = \frac{F^2}{A^2k^2}$$

The total change in length dl will be the sum of all the second order infinitesimals or

$$dl = \int \frac{F^2}{A^2k^2}\,dx$$

$$= \frac{F^2}{\pi^2 k^2}\int\frac{dx}{(a^2+b^2x^2)^2}$$

$$= \frac{2F^2}{\pi^2k^2}\left[\frac{x}{(a^2+b^2x^2)2a^2} + \frac{1}{a^3b}\tan^{-1}\frac{b}{a}x\right]$$

where x is one-half L_o, the necked length. Fig. 13 is a plot of dl for various values of b. For b=o, the hyperbola degenerates into a straight line and the specimen becomes a cylinder. It is apparent that if b=a, the half length of the specimen x, need be only 2a in order that the total extension at a given F be within a few percent of the total extension of an infinite length hyperbolic specimen (at the same F). It is also seen how much less the necked specimen with x=2a will deform than a cylinder of the same length with a cross-sectional area a^2 (the minimum area of the neck).

The important point is that the same functional relationship will be obtained for the load extension curves of a specimen of uniform cross-sectional area (b=o) as for a necked specimen (b≠o) except for a different multiplication constant. This is illustrated graphically for the case x=3, b=$^a/2$ in Fig. 14.

The above discussion is not strictly correct because, in reality, the lines of principal stress in the necked specimen are not parallel to one another except at the minimum cross-section. An exact treatment using laws of elasticity would be too cumbersome and it is felt that the correction to the above formula would be small. At large x, where stress lines deviate most from parallelicity, the total extension is very small.

FIGURES

Figure 1	Schematic Representation of Apparatus used to measure the "yield" of small specimens.
Figure 2	Stress-Strain Curves for the series of large crystals. All specimens were stressed to the same value: 580 psi.
Figure 3	Typical Interferogram from surface of a large specimen. A lightly silvered segment of a sphere is used as the reference mirror.
Figure 4	Load-Extension Curve for Specimen MG-M. Average diameter at neck = 450 microns. Cross sectional area at neck = 0.157 mm^2 "Yield Stress" $= \frac{250}{0.157} = 1600$ gms/mm^2
Figure 5	Load-Extension Curve for Specimen MG-S. Average diameter at neck = 214 microns. Cross sectional area at neck = 0.036 mm^2 "Yield Stress" $= \frac{120}{.036} = 3300$ gm/mm^2
Figure 6	Load-Extension Curve for Specimen MG-W. Average diameter at neck = 160 microns. Cross sectional area at neck = 0.026 mm^2 "Yield Stress" $= \frac{120}{0.026} = 4600$ gm/mm^2
Figure 7	Plot of log "Yield Stress" against log of Cross sectional area.
Figure 8	Orientation of Specimens.
Figure 9	Data obtained by Makin on Cadmium crystals.
Figure 10	Appearance of Typical Small Specimen after Polishing.
Figure 11	Range of neck shapes approximated by hyperbolas.
Figure 12	Plot showing how necked specimens having various shapes (See Fig. 11) would elongate at a given load. The curves show that for the hyperbolic shapes there is a maximum elongation regardless of length of the specimen.
Figure 13	Curves illustrating that the functional relationship between load and elongation is not changed (except for a constant factor) when a necked specimen is used instead of a specimen of uniform cross sectional area.

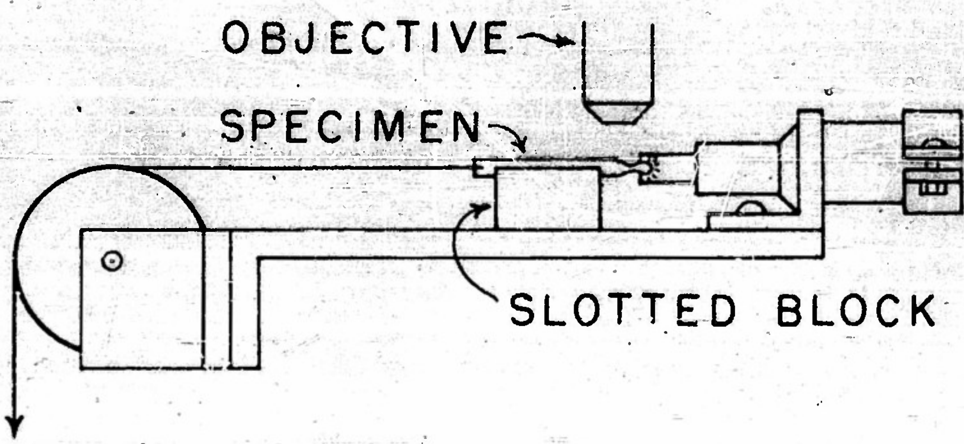


Fig. 1

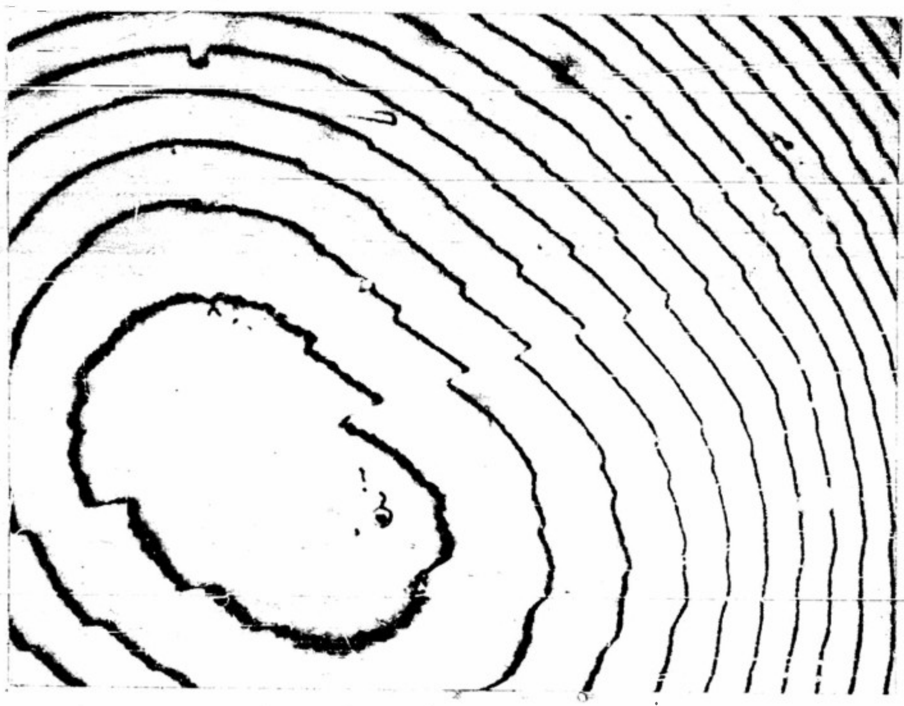


Fig. 3

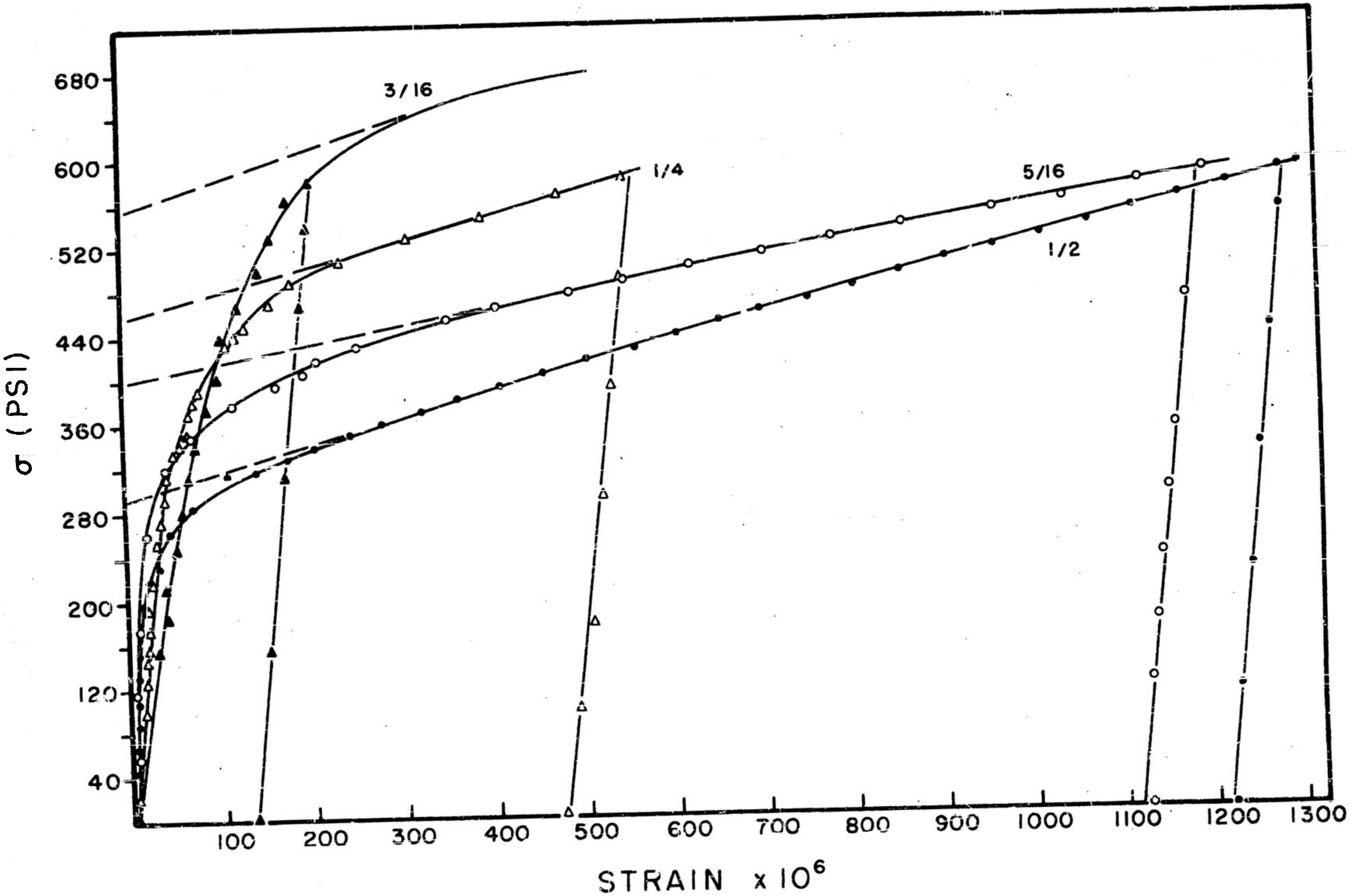
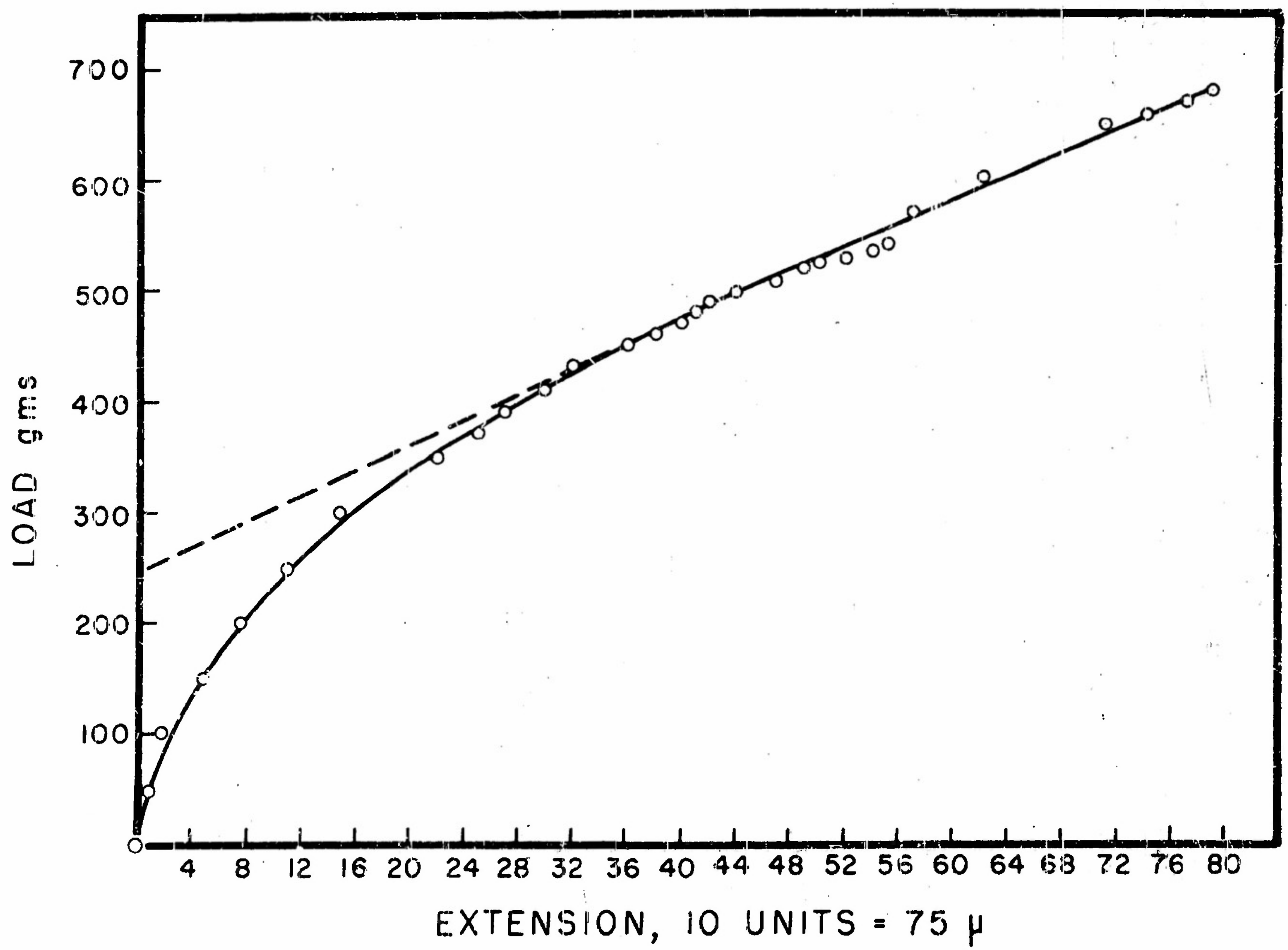

3/16
1/4
5/16
1/2
σ (PSI)
680
600
520
440
360
280
200
120
40
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
STRAIN x 10⁶

Fig. 2



700
600
500
400
300
200
100
LOAD gms
4 8 12 16 20 24 28 32 36 40 44 48 52 56 60 64 68 72 76 80
EXTENSION, 10 UNITS = 75 μ

Fig. 4

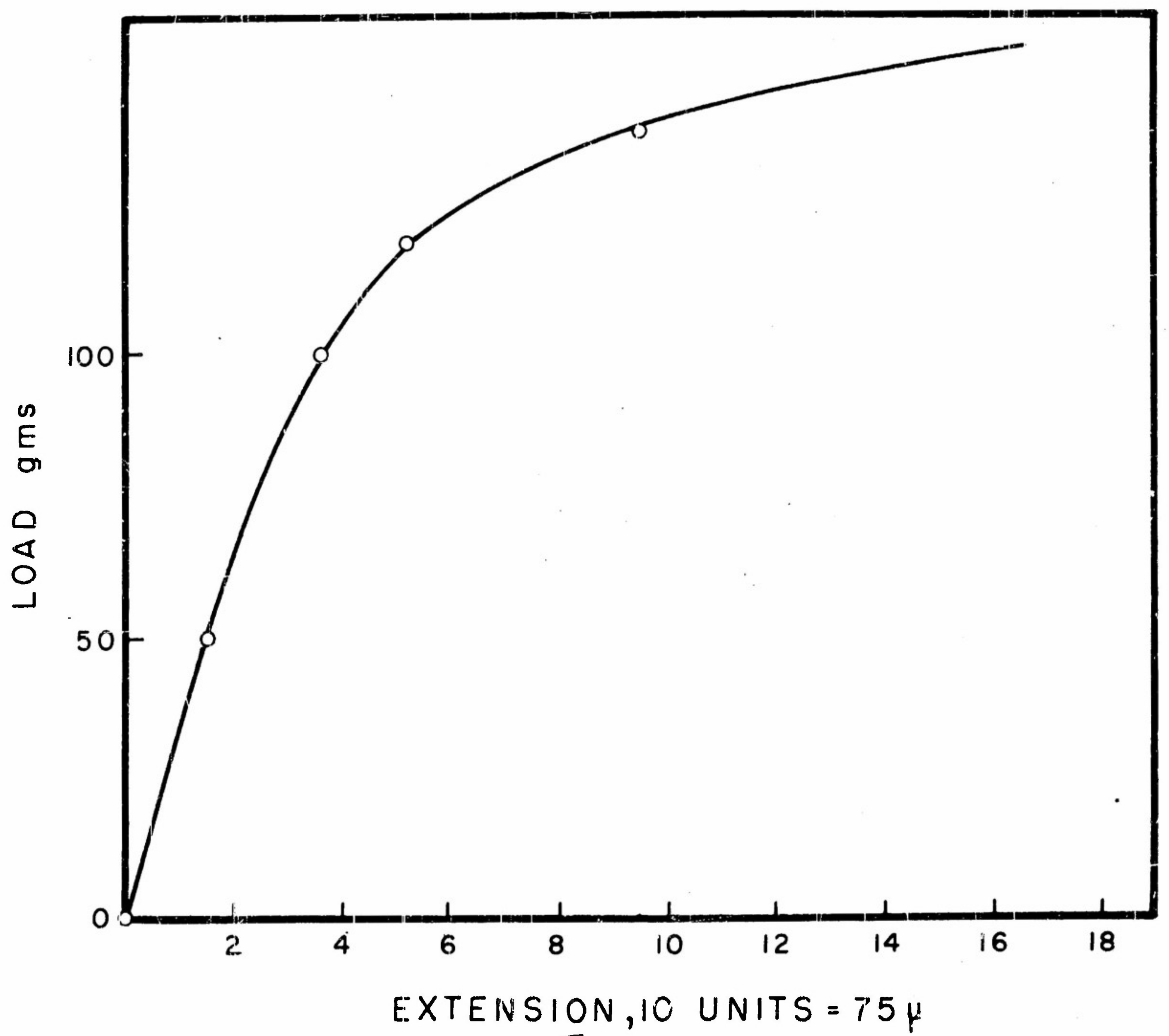


Fig 5

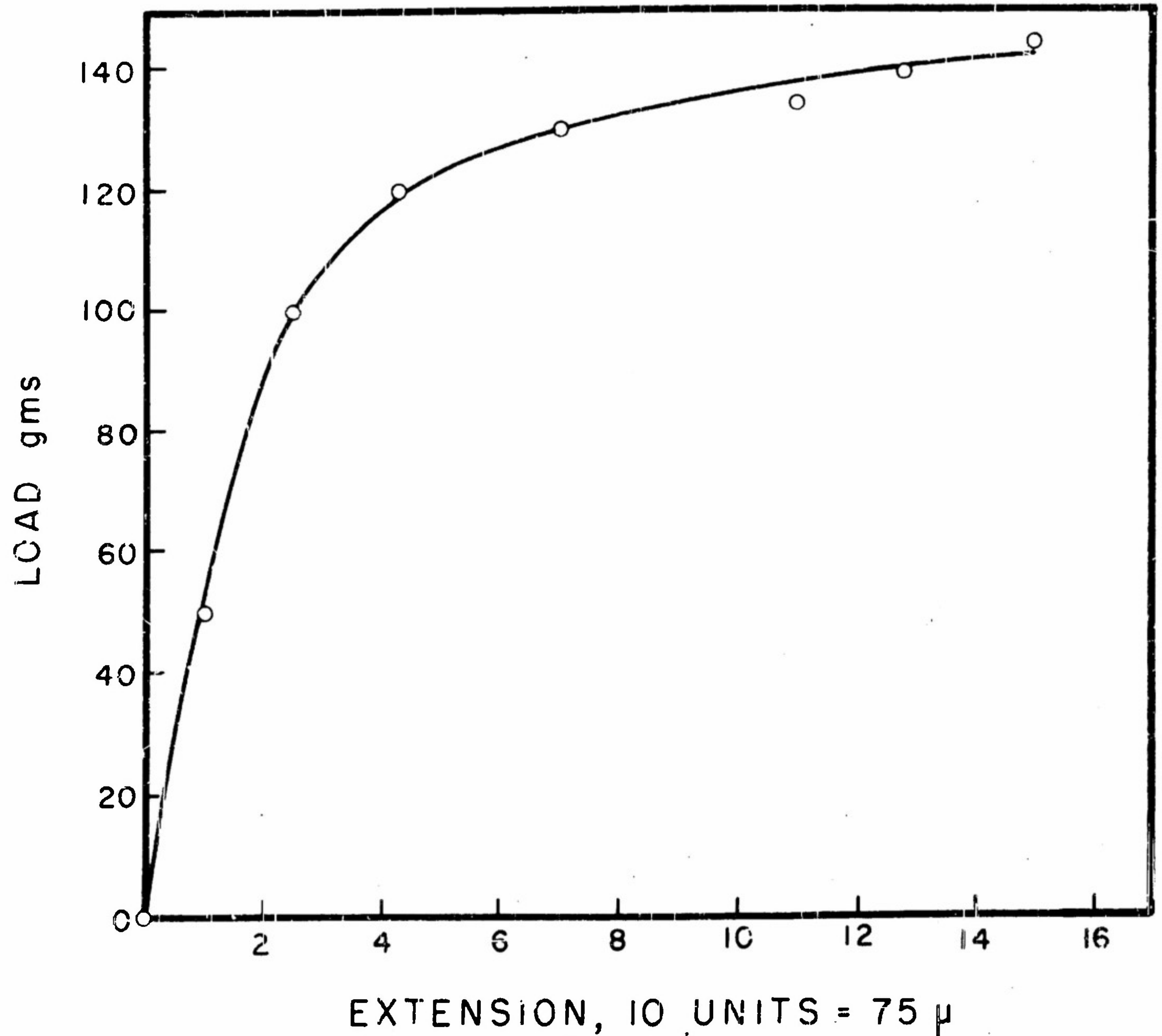
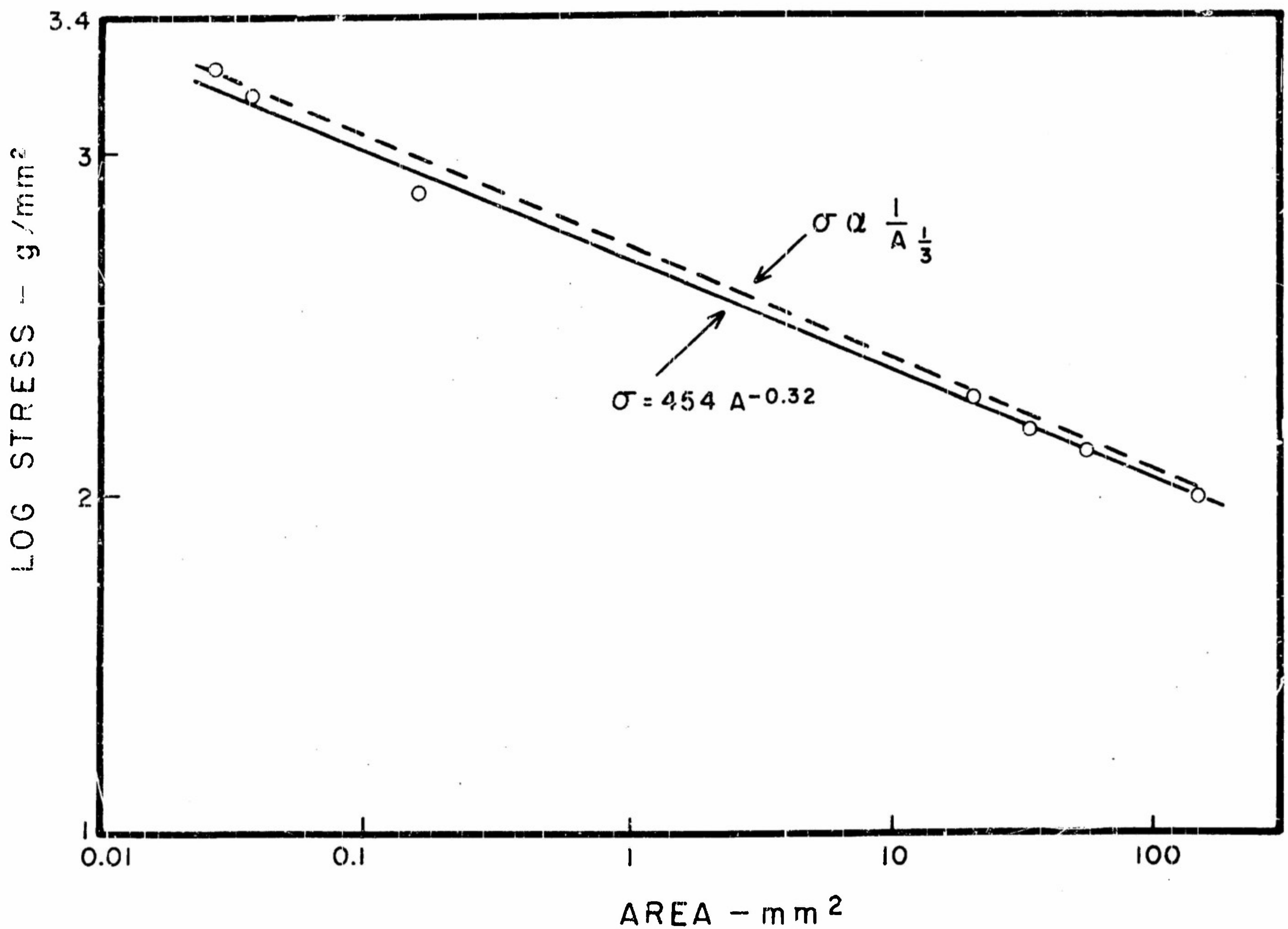

LOAD gms
0
20
40
60
80
100
120
140
2
4
6
8
10
12
14
16
EXTENSION, 10 UNITS = 75 μ

Fig. 6



3.4
3
2
1
0.01
0.1
1
10
100
LOG STRESS – g/mm²
AREA – mm²
$\sigma \propto \frac{1}{A^{\frac{1}{3}}}$
$\sigma = 454\ A^{-0.32}$

Fig. 7

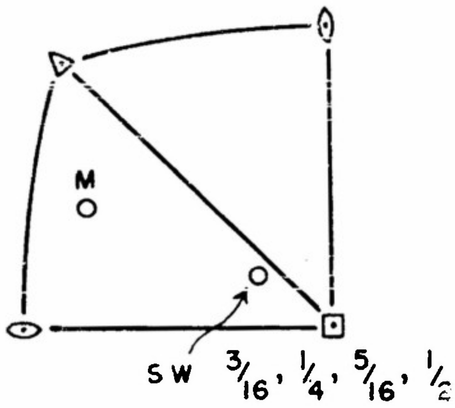

M
SW 3/16, 1/4, 5/16, 1/2

Fig. 8

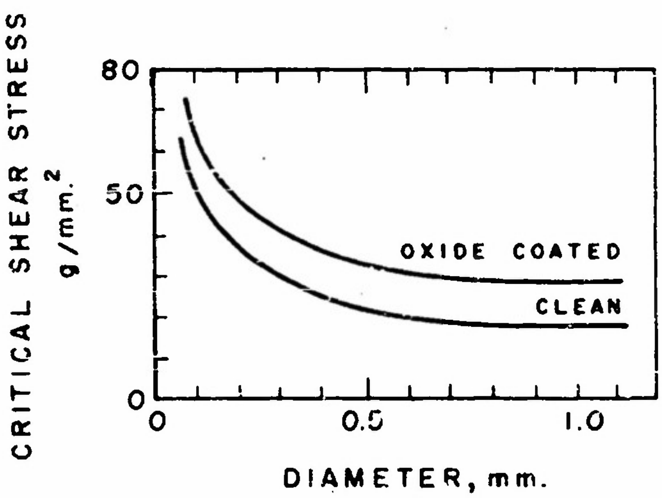

CRITICAL SHEAR STRESS
g/mm.²
80
50
0
OXIDE COATED
CLEAN
0
0.5
1.0
DIAMETER, mm.

Fig. 9

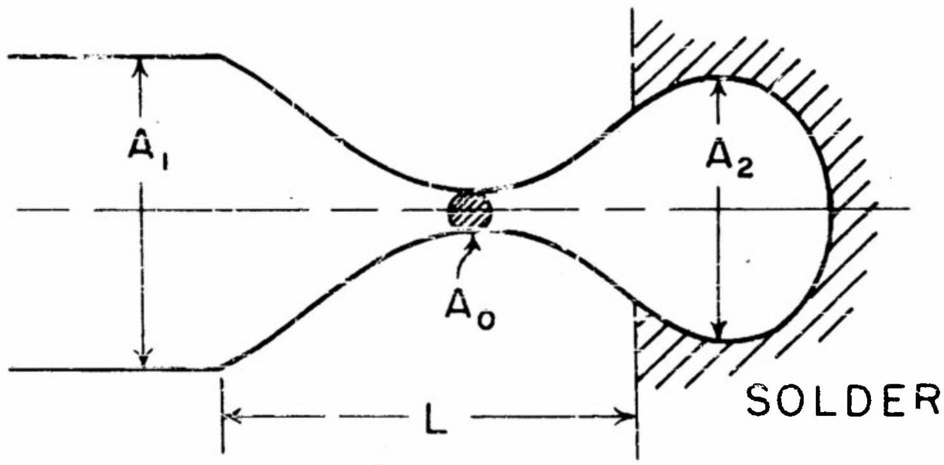


Fig. 10

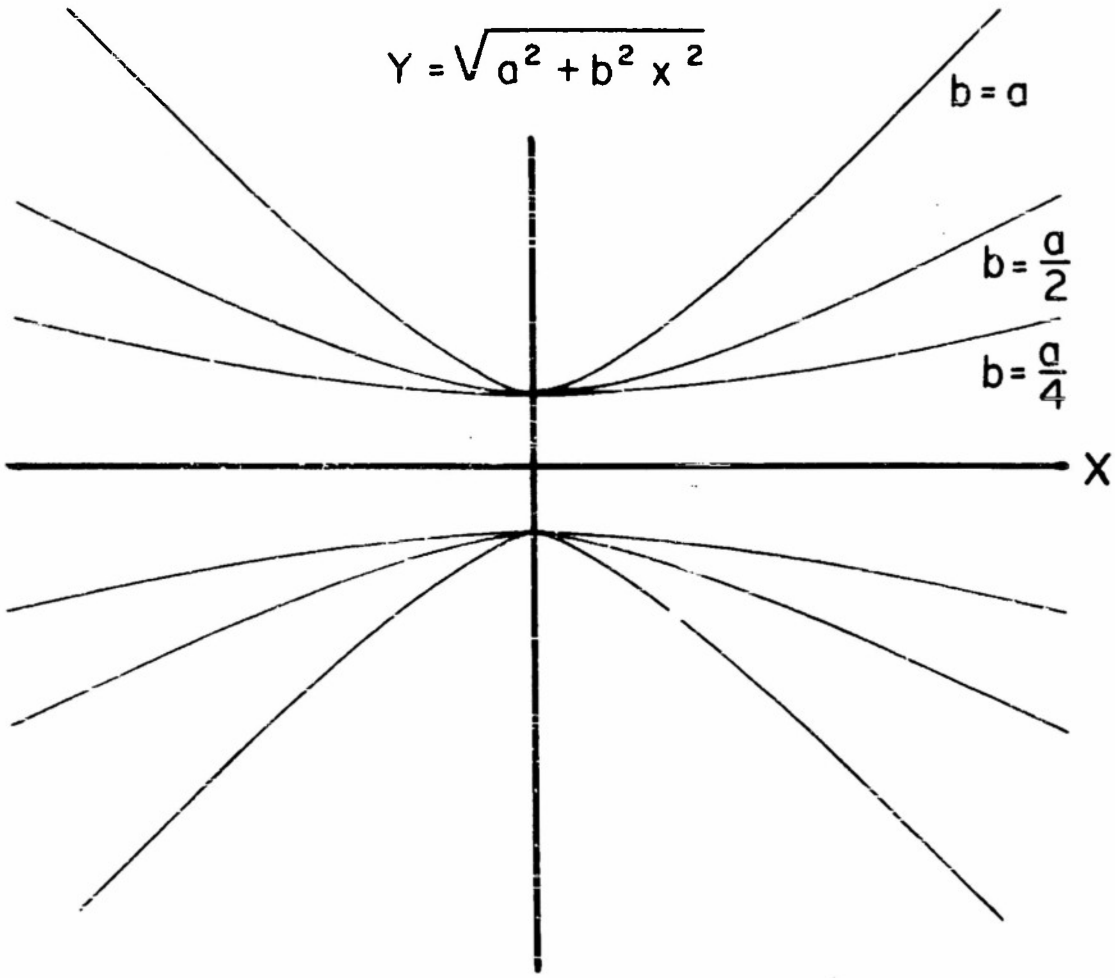


Fig. 11

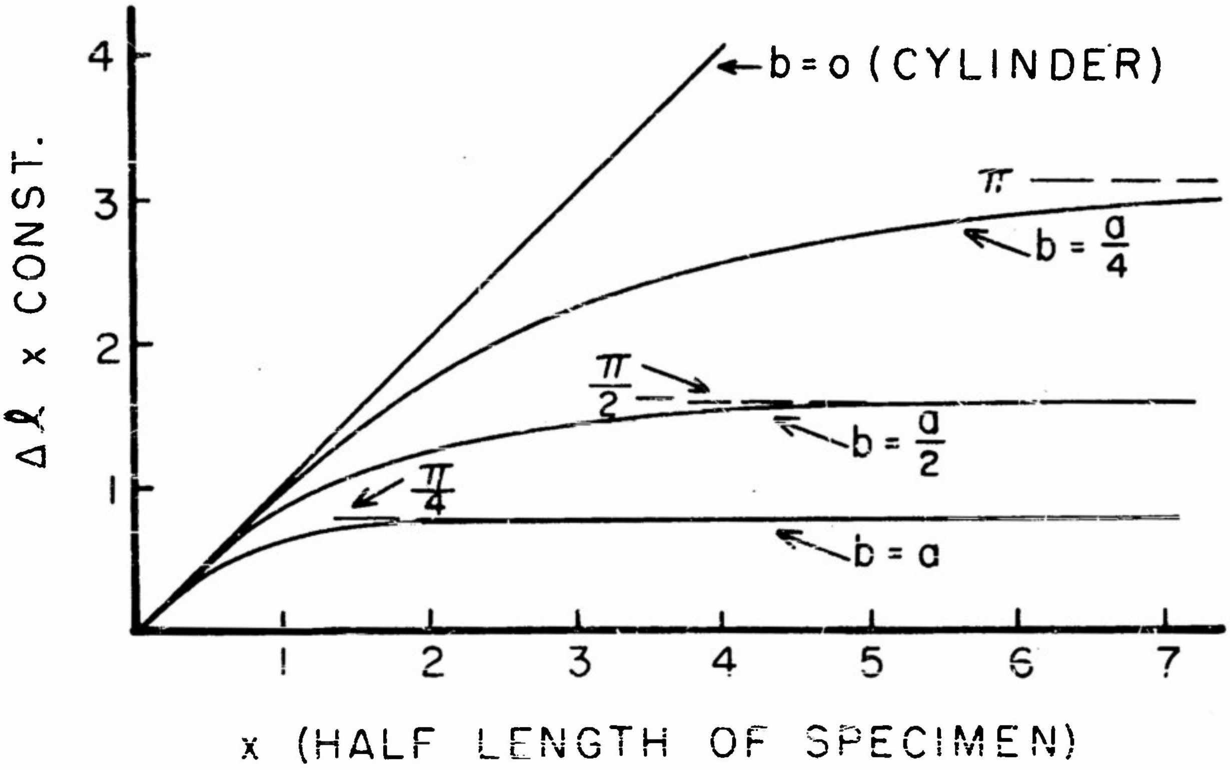

4
3
2
1
1
2
3
4
5
6
7
Δℓ x CONST.
x (HALF LENGTH OF SPECIMEN)
b = o (CYLINDER)
π
b = a/4
π/2
b = a/2
π/4
b = a

Fig. 12

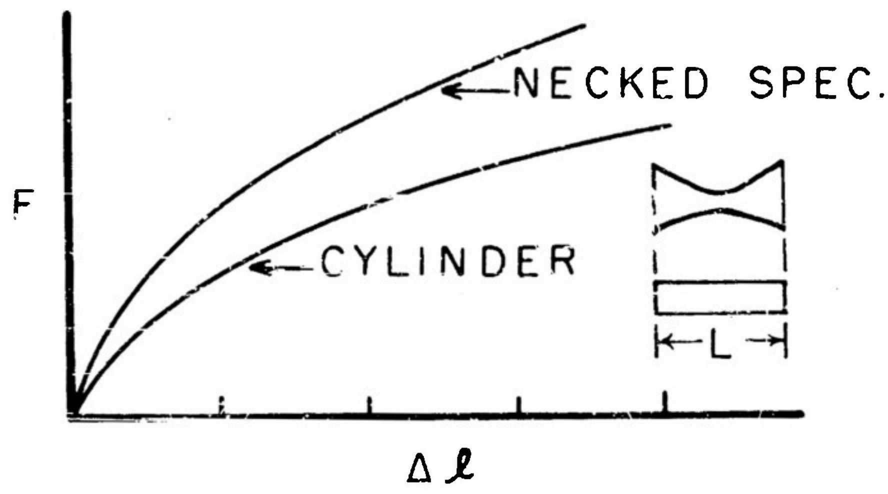

NECKED SPEC.
CYLINDER
F
L
Δℓ

Fig. 13